Spectrum imaging: the future of quantitative X-ray mapping?

I M Anderson

Oak Ridge National Laboratory, Metals & Ceramics Division, PO Box 2008, Oak Ridge TN 37831-6376 USA

Electron probe microanalysis (EPMA) has just celebrated its fiftieth anniversary and continues to be regarded as the most established and reliable technique for the elemental microanalysis of materials. As EPMA enters its next fifty years, developments in instrumentation and computing power may inspire the replacement of traditional wavelength-dispersive X-ray spectrometers (WDS) with a combination of energy-dispersive X-ray spectrometers (EDS) that would allow considerable flexibility for quantitative microanalyses, especially for quantitative elemental mapping. In particular, new high count-rate EDS detectors [1] promise to allow rapid X-ray mapping, and new high spectral-resolution detectors [2] would allow X-ray lines that exhibit strong overlaps at the resolution of conventional EDS detectors to be quantified, with excellent sensitivity. Although the substitution of EDS for WDS in X-ray mapping would involve slightly different practical challenges for quantitation, many of the fundamental strengths of the field of EPMA would still apply, most notably the fundamental notion of using suitable standards and the well established methods for atomic number, absorption and fluoresence corrections.

EDS provides many inherent advantages relative to WDS. Because the spectrum is acquired as a whole, the relevant signal is acquired most efficiently, minimizing acquisition time and beam damage, and correlations among variations in spectral features and thus elemental concentrations are unambiguous because of the parallel acquisition. In addition, quantitative microanalysis of rough surfaces or particles, which comprise an increasing fraction of important microanalytical problems, becomes practicable with EDS because of the common viewpoint for all spectral features [3]. Finally, the parallel collection of EDS allows spectrum imaging to be performed, where a full spectrum is acquired at each pixel in a two-dimensional array, providing a comprehensive characterization of a signficant sampling of the microstructure [4, 5]. Spectrum imaging provides a paradigm that combines the best features of the current paradigms in electron optical characterization for imaging, with its emphasis on contrast production, and analysis, with its emphasis on quantitation.

One factor that has limited the practicality of spectrum imaging is the large size of the raw data files so produced. Typically, digital images acquired in the scanning electron microscope are comprised of ~1 M pixels; EDS spectra are typically comprised of ~2 k channels, with 2 bytes per channel. A spectrum image combining these parameters would comprise a few Gbytes of data! Accordingly, a variety of “data mining” methods are being explored to extract the relevant information from these large data files [6]. An illustration of two such analysis methods, applied to a spectrum image of a cross-sectioned computer chip, is shown in Fig. 1: (a) linear multivariate statistical analysis (MSA) [6-8] and (b) multiple linear least squares (MLLSQ) fitting to a series of spectra representing all distinct phases in

the chip microstructure. The bright regions in Fig. 1 show the silicon dioxide dielectric in the computer chip. The 200 × 150 pixel spectrum image was acquired at 4 kV accelerating voltage and 100 nm per pixel; experimental details are given elsewhere [5]. The spatial resolution for these operating conditions was previously determined as being ~160 nm [7]. MSA is a robust analysis technique, requiring no a priori input from the analyst, is suitable for automation, but is relatively time consuming and memory intensive. In addition, the raw MSA components contain information about a variety of phases; the variety of gray levels exhibited by different features in Fig. 1a is a result of this mixing. In contrast, the MLLSQ component in Fig. 1b represents a pure component: the silicon dioxide phase is white, and all other features of the microstructure are black, to within the noise level of the data. Although the MLLSQ method requires operator input in the form of basis spectra, no iterative operator intervention is required in the analysis, and MLLSQ could be performed "on the fly" during acquisition. A suitable combination of the MLLSQ and MSA method may provide an optimum solution for spectrum image analysis and display, with the MLLSQ identifying the major phases and MSA being used to analyze spectra that do not conform to the basis initially chosen by the operator.

References

[1] Strüder L, Lechner P, Leutenegger P and Schülein T 1998 Microsc. Microanal. **4** (Suppl. 2: Proceedings), 176
[2] Wollman D A et al. 1996 Proc. Microsc. Microanal., p. 488; 1997 J. Microsc. **188**, 196
[3] Small J A, Armstrong J A, Bright D S and Thorne B B 1998 Microsc. Microanal. **4** (Suppl. 2: Proceedings), 184
[4] Jeanguillaume C and Colliex C 1989 Ultramicrosc. **28**, 252
[5] Anderson I M 2000 Microsc. Microanal. **6** (Suppl. 2: Proceedings), submitted
[6] Bonnet N 1998 J. Microsc. **190**, 2
[7] Anderson I M 1999 Microsc. Microanal. **5** (Suppl. 2: Proceedings), 318
[8] Kotula P G et al. 1999 ibid., 806

Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

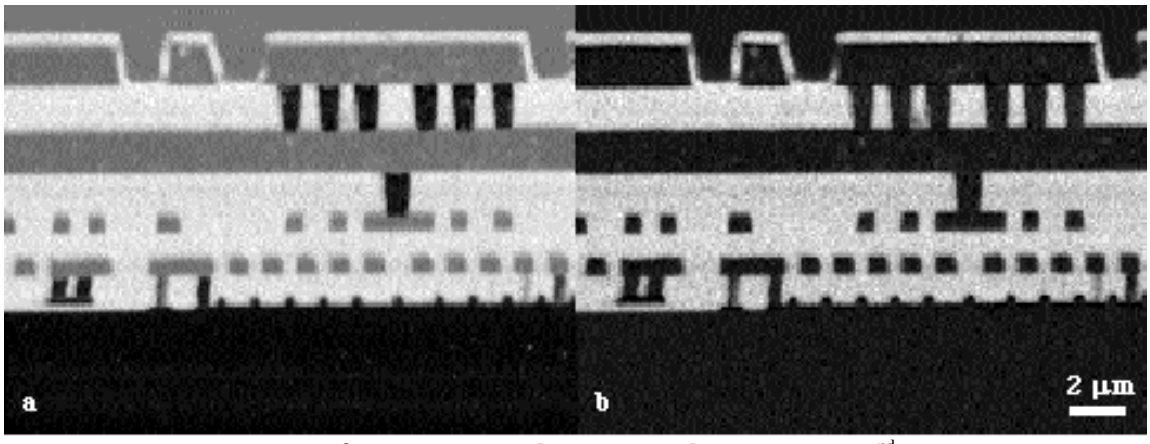

Fig. 1 – Spectrum image of cross-sectioned computer chip: (a) MSA 2nd component image with positive loading on O-K; (b) MLLSQ component image for silicon dioxide spectrum.